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FLAME RESISTANT ELASTOMERIC POLYMER DEVELOPMENT

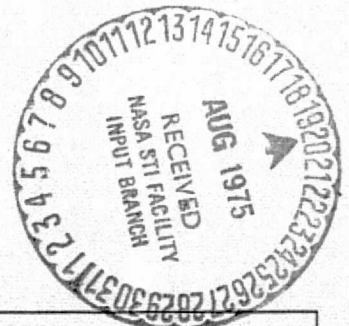
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ABSTRACT

From 1973 through 1975 our efforts have been directed toward the development of a series of new elastomeric products for use in the space shuttle program. The objectives of the program were to improve the properties of the elastomers that were used in previous programs and to evaluate the possibility of using lower-cost general purpose polymers. New and products based on the fluorinated elastomers, urethanes, and neoprenes were produced.

One of the major achievements of the program was the development of compounds that would not burn in a 70% oxygen, 30% nitrogen atmosphere and that would produce very little smoke if ignited. These compounds met toxicity requirements and were qualified at White Sands. All the compounds could be fabricated by conventional means and they should be adaptable to large-scale production.

Various products were fabricated and processed on conventional processing equipment; these were evaluated at Arthur D. Little and at NASA, Houston. These products included:

- Foams based on fluorinated rubber flame-retarded compounds with a density of 20-30 pounds/cubic foot for use as padding and in astronauts' helmets.
- Foams based on urethane for use in instrument packaging in the space shuttle. Two types were produced: one from an untreated foam for use in standard atmospheres; the other a conventional foam treated with a special compound that contained fluorinated elastomer to provide non-burning products in atmospheres of 30% oxygen, 70% nitrogen. These were all low-density foams with a density of 2 to 6 pounds/cubic foot.
- Flexible and semi-rigid films of fluorinated rubber and neoprene compounds that would not burn in a 70% nitrogen, 30% oxygen atmosphere and in a 30% nitrogen, 70% oxygen atmosphere, respectively, for use in packaging or in laminates.
- Coated fabrics which used both nylon and Kelvar fabric substrates, coated with either fluorinated or neoprene polymer compositions to meet specific levels of flame retardancy. These are being evaluated for use in firefighters' suits and in astronaut clothing. Fifty yards of the nylon-neoprene combination was prepared and is to be made into suits.

- Paints for use on metal and other substrates to provide flame resistant coatings.
- Adhesives for bonding a variety of products. These adhesives were based on either the fluorinated polymers or the neoprene compositions.
- A flame resistant hose that resists hydraulic fluids and petroleum-based products and can be used in a 70% oxygen; 30% nitrogen atmosphere.
- Compounds for molding lens covers at NASA. These compounds were based on the low-smoke fluorinated rubber compounds that would not burn in 100% oxygen atmosphere.

Under an extension of the contract an experimental program is under way to utilize the technology developed for elastomers to produce rigid structures that have equivalently high flame retardancy.

I. INTRODUCTION

The objective of this program has been to develop elastomeric products for use in the space shuttle program. The overall program has been conducted under three programs:

1. Contract NAS 9-10424

Flame retardant urethane polymers were synthesized and additives were incorporated in fluorinated and urethane polymers to impart flame retardancy. Compositions that were self-extinguishing in the 70% oxygen, 30% nitrogen atmosphere were made into fibers and incorporated into fabric structures where the elastomeric fibers were wrapped with non-elastic PBI, Nomex and beta-Fiberglass to give non-flammable elastomeric structures.

2. Contract NAS 9-12815

The strength of the elastomeric fibers was improved substantially and manufacturing techniques were refined. Quantities of fiber were prepared and power net fabrics were made that were satisfactory for sleep-monitoring caps and other applications in the spacecraft.

3. Contract NAS 9-13979

Utilizing the technology developed in the above contracts, we adapted the elastomeric compositions to other products - such as foams, paints, adhesives, fabric coatings, hose, etc. - for use in the air shuttle program.

II. BACKGROUND

In the program for 1970-1971, which is covered in our report dated March 1971, various urethane-type polymers were synthesized, and many had better flame resistance than spandex. However, flame resistant fibers were produced most efficiently by incorporating halogenated additives into urethane or fluorocarbon elastomers. Some of the resultant fibers were not flammable even in a 100% oxygen environment.

Various flexible polyurethane structures containing halogen were synthesized from polyesters derived from aliphatic or aromatic polyols and dibasic acids. Aliphatic halide structures could not be used because they are unstable at the required reaction temperatures, giving off hydrogen halide which hydrolyzes the ester linkages. In contrast, halogen-containing aromatic polyols are stable and lead to satisfactory products.

The most promising composition, a brominated neopentyl glycol capped with toluene diisocyanate, was used as a conventional diisocyanate in conjunction with hydroxy-terminated polyethers or polyesters to form elastomeric urethanes containing about 10% bromine by weight. Products made in this manner will not burn in air, have an oxygen index value of about 25, and have tensile strength values of about 5000 psi at 450% elongation.

The most efficient additives for imparting flame retardancy to spandex urethanes are aromatic halides and the most effective of these are the bromide compounds. Various levels of flame retardance were achieved, depending on the levels of additives used. Compositions were prepared and tested to meet the following specifications:

a. 31% Oxygen/69% Nitrogen at 10 psia

These urethane compounds have tensile strengths of 1500-2000 psi and elongations of 300-450%. They contain 20-30% urethane with the remainder flame-retardant additives (hexabromobenzene in combination with trisbromochloropropyl phosphate as a flame-retardant plasticizer).

b. 70% Oxygen/30% Nitrogen at 6.2 psia

These urethane compositions have tensile strengths of 400-900 psi, elongations of 250-400%, and oxygen index values of 75-85. They are composed of about 5% urethane, 20-30% fluorinated polymer, and 65-75% hexabromobenzene/trisbromochloropropyl phosphate.

Although it was possible to achieve the necessary flame retardancy with the urethanes, the physical properties were so inferior that serious problems would be encountered in making fabrics with the necessary recovery and strength characteristics. For these reasons a fluorocarbon elastomer com-

pound was developed that provided better nonflammability and higher strength. The major portion of our work has been with this material and the approved product was based on a fluorocarbon elastomer.

In the second phase of the program, carried out in 1971 and early 1972, the physical properties of the elastomeric fibers were improved and a limited number of fabrics were produced from these fibers in combination with fiberglass, Nomex and PBI fibers. To achieve these results, we had to evaluate a large variety of flame-retardant additives and prepare enough of the fabrics to indicate the type of structure that would be required to obtain properties nearly equivalent to those of standard spandex fiber fabrics. During this phase, we prepared films and fibers, as well as knit structures, that met the flame-retardancy requirements and that have physical properties approaching those of spandex fabrics. We achieved equivalent physical properties by increasing the amount of elastomeric material in the fabric.

The third phase, carried out in 1972 and early 1973, resulted in a substantial improvement in physical properties and manufacturing techniques. Properties were especially improved when a standard curing system was replaced by a unique cure based on peroxide and maleimide. This change made the compound easier to process, and provided the elongation and strength necessary to make a satisfactory fiber. Rates of production and fiber characteristics were improved by the use of a new 1" extruder with a longer barrel, the use of a modified die with a larger number of orifices, the use of an improved take-up device, and improvements in curing procedures.

PBI fibers were selected for wrapping the elastomer fibers. Structures of power net were developed by John Somak, Inc., in cooperation with us. One of these was selected and 50 pounds of material was extruded for use in making sleep monitoring caps, and other crew equipment and spacecraft applications in the Skylab program.

The fourth and fifth phases, carried out in 1973 through 1975 (covered in this report), have resulted in a number of products for different space shuttle programs. These products were designed to meet a variety of flame retardancy requirements ranging from not burning in air to not burning in a 100% oxygen atmosphere. New polymers were investigated and compounds and products were prepared from the fluorinated elastomers, neoprene and urethane. Smoke levels of the fluorinated polymers were reduced to acceptable limits and at the same time the flame retardancy requirements were met. Highly successful products were developed and are in use in the space shuttle program. These include compounds for lens covers, foams for use as a lining in space helmets, foams for packaging instruments, and coated fabrics for astronaut suits and firefighter suits.

Other products were also prepared to demonstrate that they could be made by conventional commercial processes. These include paints, films, and adhesives.

III. PROPERTIES AND COMPOUNDING

This section reviews the steps that were taken to improve flame retardancy and physical properties and to reduce smoke emission. Specific compounding details that were required to provide properties for the various end uses are discussed in Section IV.

A. POLYMERS

In our program we used three polymers: fluorinated elastomers, urethanes, and polychloroprene. Each type of polymer was used in one or more of the end products, depending on the particular properties and level of flame retardancy that were required. Other types of elastomeric polymers are not suited for use in this program either because of very high flammability characteristics or because of very high smoke emission. The characteristics of the polymers used are discussed below.

1. Fluorocarbon Elastomers (Viton)

Of all the elastomers, this material has the highest level of inherent flame retardancy because of the fluorine in the molecule. Compounds of Viton were successfully used in the end uses under consideration; these compounds had a low smoke emission and high levels of flame retardancy (yielding oxygen index values between 35% and 100%, depending upon filler loading).

The major disadvantage of the fluorinated polymers is their cost--\$10.00/pound. However, this cost is offset to some extent by the use of inexpensive fillers and flame retardants.

Fluorinated elastomers may be fabricated into finished products by calendering, extrusion, injection molding and compression molding, and may be applied from solvent solution for use as cements, spread coatings, or paints. Paints and adhesives capable of being cured at room temperature may be prepared.

Compounds of the material have been forwarded to NASA and a number of them have been approved with respect to flammability, low smoke emission, and low toxicity.

2. Chloroprene (Neoprene)

Neoprene, like the fluorinated elastomers, has a built-in flame retardance because it contains chlorine in its molecule. Consequently, without the addition of special flame retardants it has an oxygen index value of 25 to 30. However, by adding additional flame retardant materials, it can be made to meet the 30% oxygen, 70% nitrogen specification required for certain NASA applications. At about 35¢/pound, chloroprene

is relatively inexpensive, and like the fluorinated elastomers, it can be calendered, extruded, injection molded and compression molded, or be used as a solvent solution in cements or spread coatings.

Because of the chlorine present in the chloroprene molecule, the product tends to produce a high level of smoke. In this respect, it is much worse than the fluorocarbon elastomers, which are fairly low smoke producers.

3. Urethanes

The flexible urethanes of either the polyether or polyester type burn readily if not modified, and if they are to be used successfully in applications at NASA, flame retardant must be added. Because the urethanes are ideally suited for making foams over a wide density range from 1 1/2 pounds/cubic foot and up, we used them to make foams for various NASA applications.

Instead of incorporating flame retardants directly into the composition, we found it more practical to saturate a commercially produced urethane foam with a flame retarded fluorocarbon elastomer compound. In this fashion, we were able to produce foams that would meet the 30% oxygen, 70% nitrogen requirement for use in packaging applications.

4. Ethylene Propylene Diene (EPDM)

EPDM has many excellent properties for NASA's application because it has excellent resistance to ozone and aging, and because it produces very little smoke on burning. The polymer, however, has proved to be very difficult to make flame retardant. Neither hydrated alumina nor ammonium polyphosphate increased the flame retardancy to acceptable levels, and it was necessary to use halogenated materials. These halogenated compositions produced high levels of smoke so the compounds were no better in this respect than neoprene. Because of this problem we did only a limited amount of work with this polymer and instead devoted our efforts to the polymers whose chemical structure makes them inherently more flame retardant. Table III-1 shows the significant properties and costs of these polymers.

B. SMOKE RETARDANTS

The major objective of the investigation originally had been to produce products with the required level of flame retardancy. The emphasis at this stage was directed to the development of compounds that had both a high level of flame retardancy and at the same time would produce minimal levels of smoke. Among the base polymers, the low smoke producers are EPDM and the fluorocarbon elastomers. The conventional general purpose elastomers are high smoke producers and, in addition, very difficult to compound to the flame resistance levels required. After examining the physical properties and burning characteristics of

TABLE III-1

CANDIDATE POLYMER SYSTEMSFLEXIBLE TYPE

<u>Material</u>	<u>L.O.I.*</u>	<u>Tensile Strength</u> (psi)	<u>Elongation</u> (%)	<u>Operating</u> <u>Temperature</u> (°F)	<u>Cost Per</u> <u>Pound</u> (£)
<u>Fluorinated Elastomers</u>					
Polymer	40	2000-3500	300-450	400	10.00
Compounded**	40-100	2000-4000	300-600	400	5.00
<u>Polyurethane</u>					
Polymer	<30	6000	300-600	250	.50-2.50
Compounded**	30-60	2000-4000	300-600	250	.95-2.00
<u>Chloroprene</u>					
Polymer	25	3000-4000	300-600	250	.45
Compounded**	25-55	2500-3000	300-600	250	.65
<u>Ethylene Propylene Rubber</u>					
Polymer	<20	2000-3000	300-600	250	.35
Compounded**	20-30	1500-2500	300-600	250	.80

* In order to pass NASA 30% O₂ and 70% N₂ flammability test, a limiting oxygen index of 45-50 is probably required.

** Arthur D. Little, Inc., estimates to meet NASA flammability requirements.

all the elastomers, we selected the fluorinated elastomers, the urethanes, and neoprene as the most suitable for modification. Although EPDM produces very little smoke, it burns so readily that it is virtually impossible to find a compound to meet the flame retardancy requirements of NASA without resorting to halogenated materials which in themselves are high smoke producers.

The most effective flame retardants are compounds that contain either chlorine or bromine, but these produce high levels of smoke. In our work, therefore, we used two approaches. The first was to use smoke suppressants with the polymer and halogen-containing flame retardant to reduce the level of smoke. The second was to develop flame retardant compounds that did not use halogen-containing compounds. The latter proved to be the most successful approach.

Table III-2 lists the additives that were evaluated with the various polymers along with decabromobiphenyl flame retardant. The additives were selected on the basis of literature references and recommendations from various industrial sources. The most interesting result of this study was that hydrated alumina both increased the flame resistance and reduced the smoke. None of the other materials reduced smoke effectively. Tables III-3 through III-6 show the specific compositions that were used.

We evaluated a series of flame retardants with Viton fluorocarbon elastomer. A list of these and their exact composition is shown in Table III-7. Of the materials tested for reducing smoke, the most effective were flame retardants that inherently were not smoke producers.

The best of these low-smoke flame retardants were hydrated alumina and samples of Viton filled with it were sent to NASA for an evaluation of their burning characteristics. In the silicone igniter tests in a 31% oxygen, 69% nitrogen atmosphere at 10 psia, these samples tended to keep burning after they were ignited because of afterglow.

When this problem became evident, various glow retardants were added to a fluorinated rubber composition which contained hydrated alumina. Since the afterglow would present a problem in the Space Shuttle program, we evaluated a series of inorganic additives which could be added to the Viton and hydrated alumina to reduce the afterglow. We evaluated the samples in a 40% oxygen, 60% nitrogen atmosphere and determined that samples containing ammonium polyphosphate or zinc borate had less afterglow than hydrated alumina alone. The materials which were evaluated are shown in Table III-8.

Since both ammonium polyphosphate and zinc borate reduced the afterglow in Viton formulations containing hydrated alumina, it seemed possible that combinations might provide a synergistic effect at certain levels. However, such was not the case. (See Table III-9.)

TABLE III-2

SMOKE SUPPRESSANTS

<u>Polymer</u>	<u>Flame Retardant</u>	<u>Additive</u>	<u>L.O.I.</u>	<u>Effect on Smoke Level</u>
Viton	Dacabromobiphenyl	Zinc Phenyl Sulfonate	95	No effect
Neoprene	Ammonium Bromide	Ferrocene	36	No effect
Neoprene	Hydrated Alumina	-	-	Slight improvement
SBR	Hydrated Alumina	Ferrocene	29	No effect
EPDM	Hydrated Alumina	Ferrocene	28	Relatively low smoke; additives ineffective for smoke reduction.
"				
Viton	None	Lead Phosphite	40	No effect
Viton	None	Vinylidene Fluoride	35	Slight improvement
Viton	Antimony Oxide	Hydrated Alumina	35	Slight improvement
Viton	Hydrated Alumina (100 parts)	-	50	Substantial improvement
Viton	None	Sodium Aluminum Fluoride	37	No effect
Viton	None	Basic Lead Carbonate	38	No effect
Viton	None	Zinc Borate	35	No effect
Viton	None	Potassium Sulfate	45	No effect

(reduces afterglow)

TABLE III-3

FLUORINATED POLYMER (VITON)

<u>Material</u>	<u>Parts By Weight</u>			
<u>Code #106</u>	<u>A</u>	<u>B</u>	<u>C</u>	<u>D</u>
Viton B	100	100	100	100
Lead Oxide	15	15	15	15
Maleimide	4	4	4	4
Benzoyl Peroxide	4.5	4.5	4.5	4.5
Decabromobiphenyl	100	100	100	
Zinc Phenolsulfonate		5.0	10	
Cure minutes @ 270°F	60	60	60	60
Post Cure @ 450°F hours				15
Oxygen Index L.O.I.	100	95	95	60
Smoke	Medium	Low	Medium	Low
<u>NASA Tests</u> *				
Burning Time (seconds)	50	120	60	60
Burned Length (inches)	1	1	1	1-1/2
Smoke Density	Heavy	Heavy	Dense	Dense
Color	White	White	White	White
Odor	Strong	Strong	Strong	Strong

* Test atmosphere 10 psia 31% O₂, 69% N₂
Silicone igniter

TABLE III-4

NEOPRENE

<u>Material</u>	<u>Parts By Weight</u>		
<u>Code #108</u>	<u>A</u>	<u>B</u>	<u>C</u>
Neoprene W	100	100	100
Magnesium Oxide	4	4	4
Clay	85	85	85
Hydrated Alumina (Alcoa C-333)	85	85	85
Ammonium Bromide	-	40	40
Ferrocene	-	-	4.0
Process Oil	55	55	55
Zinc Oxide	5	5	5
Titanium Dioxide	10	10	10
Stearic Acid	6	6	6
Antioxidant	2	2	2
NA-22	0.5	0.5	0.5
Cure Condition min. @ 350°F	20	20	20
L.O.I.	26-27	36	35
Smoke	Medium	Medium	Medium

NASA Tests *

Burning Time (seconds)	Total Burn	Total Burn	Total Burn
Burned Length (inches)	.18"/sec.	.116	.125
Smoke	Heavy	Heavy	Heavy
Density	sooty	sooty	sooty
Color	Black	Black	Black
Odor**	Acid	Acid	Acid

* Test atmosphere 10 psia 31% O₂, 69% N₂
Silicone igniter

** Arthur D. Little, Inc., comment

TABLE III-5

BUTADIENE STYRENE (SBR)

<u>Material</u>	<u>Parts By Weight</u>		
<u>Code #109</u>	<u>A</u>	<u>B</u>	<u>C</u>
SBR 1506	150	150	150
Natural Rubber	15	15	15
Whiting	200	200	100
Clay	100	100	50
Hydrated Alumina (Alcoa C-333)	-	150	150
Process Oil	116	58	58
Ammonium Bromide	-	120	-
Dechlorane 604	-	-	120
Ferrocene	-	5	5
Zinc Oxide	12	12	12
Sodium Bicarbonate	15	15	15
Antioxidant	1.5	1.5	1.5
Stearic	15	15	15
MBTS	2.0	2.0	2.0
TMTD	2.0	2.0	2.0
DOTG	1.0	1.0	1.0
Sulfur	7.5	7.5	7.5
Cure Condition min. @ 400°F	2	2	2
L.O.I.	19	29	27
Smoke	Medium To High	Medium To High	Medium To High

NASA Tests*

Burning Time (seconds)	Total burn	Total burn	Total burn
Burned Length (inches)	.16	.15	.12
Smoke Density	Heavy	Heavy	Heavy
Color	Black	Black	Black
Odor	Strong	Strong	Strong

* Test atmosphere 10 psia 31% O₂, 69% N₂
Silicone igniter

TABLE III-6

EPDM

<u>ADL CODE #</u>	<u>119-B</u>	<u>119-C</u>	<u>119-D</u>
EPDM Nordel 1040	100.0	100.0	100.0
Hydrated Alumina	200.0	100.0	200.0
Zinc Oxide	5.0	5.0	5.0
Titanium Oxide	-	35.0	-
Tri-2 ethyl hexyl phosphate	25.0	-	25.0
Antimony Oxide	-	-	25.0
Dechlorane 602	-	-	50.0
EPTAC-1	2.5	-	2.5
MBT	0.5	-	0.5
Sulfur	2.5	0.3	2.5
Benzoyl Peroxide	-	2.7	-
Curing Temperature (°F)	320	290	320
Cure Time (minutes)	15	20	15
NASA Tests in Ambient Air			
Burning Time (seconds)	127	65	210
Burned Length (inches)	Burned Totally	Burned Totally	Self-exting. 1"
Rate of Propagation (in/sec)	0.04	0.07	
Smoke Density	Very Dense Smoke	Little Soot	Very Dense White Smoke
Odor		Very Dense White Smoke	Strong Odor
In 10 psia 31% O ₂ & 69% N ₂			
Burning Time (seconds)	-	-	20
Burned Length (inches)	-	-	Burned Totally
Rate of Propagation (in/sec)	-	-	0.15
Smoke Density	-	-	Very Dense White Smoke
Odor	-	-	Strong Odor

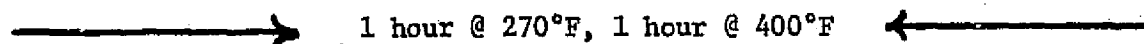
TABLE III-7

FLAME/SMOKE RETARDANTS FOR VITON

Formulation 16933-

<u>Ingredients</u>	<u>114C</u>	<u>114D</u>	<u>114E</u>	<u>114F</u>	<u>114G</u>	<u>114H</u>	<u>114I</u>	<u>115-1</u>	<u>115-2</u>	<u>115-3</u>
Viton B	100	100	100	100	100	100	100	100	100	100
Lead Oxide	15	15	15	15	15	15	15	15	15	15
Lead Phosphite ¹	15	-	-	-	-	-	-	-	-	-
Vinylidene Fluoride ²	-	15	15	15	-	-	-	-	-	-
Antimony Oxide ³	-	-	7.5	7.5	-	-	-	-	-	-
Hydrated Alumina ⁴	-	-	-	7.5	100	-	-	-	-	-
NaAlF ₆ ⁶	-	-	-	-	-	5	-	-	-	-
Potassium Sulfate	-	-	-	-	-	-	1.0	-	-	5
Basic Lead Carbonate	-	-	-	-	-	-	-	50	-	-
Zinc Borate	-	-	-	-	-	-	-	-	15	-
Maleimide	4	4	4	4	4	4	4	4	4	4
Benzoyl Peroxide	4.5	4.5	4.5	4.5	4.5	4.5	4.5	4.5	4.5	4.5

Cure Schedule


 1 hour @ 270°F, 1 hour @ 400°F

Flammability

L.O.I.	40	35	35	35	50	37	-	38	35	45
Smoke Developed	med.	low	low	med.	very low	med.	-	-	-	-

1 Dyphos, National Lead

2 Kynar, Pennsalt

3 Thermoguard W, M&I Chemical

4 Hydral 705, Alcoa

TABLE III-8

EVALUATION OF GLOW RETARDANT INORGANIC ADDITIVES

ADL Notebook #	125-A	125-B	125-C	125-D	125-E	125-F	125-G	125-H	125-I	125-J	126-L	126-M
Viton B	100	100	100	100	100	100	100	100	100	100	100	100
Lead Oxide	15	15	15	15	15	15	15	15	15	15	15	15
Hydrated Alumina			100	75	75	75	75	75	75	75	50	
Ammonium Phosphate [Phoscheck P-30]				25	--							
Zinc Borate [Firebrake ZB]					25							
Dibasic Lead Phosphate [Diphos]		10				25						
Aluminum Phosphate							25					
Phosphonitrilic Chloride								25				
Potassium			5						25		25*	
Zinc Oxide		10										
Iron Oxide			5									
Calcium Phosphate										25		
Lead Silicates												25
Maleimide [HVA-2]	4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.0
Benzoyl Peroxide	4.5	4.5	4.5	4.5	4.5	4.5	4.5	4.5	4.5	4.5	4.5	4.5
Curing Temp. (°F)	270	270	270	270	270	270	270	270	270	270	270	270
Curing Time (Minutes)	45	45	45	45	45	45	45	45	45	45	45	45
Burning characteristics in [40% O ₂ & 60% N ₂] atmosphere												
Ignition source and time (seconds)	H ₂ -15	H ₂ -15	H ₂ -15	H ₂ -15	H ₂ -15	H ₂ -15	H ₂ -15	H ₂ -15	H ₂ -15	H ₂ -15	H ₂ -15	H ₂ -15
Total flame time after ignition source is removed (seconds)	B.C. 60	B.C. 30	B.C. 30	B.C. 30	B.C. 30	B.C. 35	B.C. 60	B.C. 35	B.C. 35	B.C. 35	B.C. 45	B.C.* 45
Total burning time in- cluding after glow (seconds)	--	--	> 180	60	60	--	--	--	--	120	--	
Burned length (inches)	4	4	2 1/8	1	3/4	4	4	4	4	4	4	4
NASA Test Results in 10 psia, 31% O ₂ & 69% N ₂												
Burning time (seconds)				180	600							
Burned length (inches)				1 1/2	1 3/4							
Smoke				Medium white smoke	Dense white smoke							

* Ball Milled K₂SO₄

* B.C. = Burned Completely

TABLE III-9

SYNERGISTIC EFFECTS OF BORATES AND PHOSPHATES

ADL Code #	126-A	126-B	126-C	126-D	126-E	126-F	126-G	126-H	126-I	126-J	126-K
Viton B	100	100	100	100	100	100	100	100	100	100	100
Lead Oxide	15	15	15	15	15	15	15	15	15	15	15
Hydrated Alumina				25	50	25	25	25	50	50	50
Ammonium Phosphate [Phoscheck P-30]	25		12.5			25	12.5		25	--	12.5
Zinc Borate [Firebrake ZB]		25	12.5				12.5	25		25	12.5
Maleimide [HVA-2]	4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.0
Benzoyl Peroxide	4.5	4.5	4.5	4.5	4.5	4.5	4.5	4.5	4.5	4.5	4.5
Curing Temp. (°F)	270	270	270	270	270	270	270	270	270	270	270
Curing time (minutes)	45	45	45	45	45	45	45	45	45	45	45
Burning characteristics in 40% O ₂ and 60% N ₂											
Ignition source and time (seconds)	H ₂ -15	H ₂ -15	H ₂ -15	H ₂ -15	H ₂ -15	H ₂ -15	H ₂ -15	H ₂ -15	H ₂ -15	H ₂ -15	H ₂ -15
Total flame time (seconds)	B.C. 35	B.C. 35	B.C. 30	B.C. 35	B.C. 65	B.C. 40	B.C. 65	B.C. 45	B.C. 40	B.C. 45	B.C. * 45
Burned length (inches)	4	4	4	4	2		4	4	4	4	4

B.C. = Burned Completely

To determine the type of filler that would be most effective, we evaluated a number of formulations. From these, we selected hydrated alumina and ammonium polyphosphate. (See Table III-10.) However, compounds that contain the ammonium polyphosphate tend to produce more smoke than those that contain hydrated alumina. For this reason, we decided that a combination would be most effective--the ammonium polyphosphate for afterglow reduction, and the hydrated alumina for smoke reduction.

Tests conducted at NASA showed that a combination of 50 parts of hydrated alumina and 50 parts of ammonium polyphosphate to 100 parts of Viton provided an optimum combination of flame retardance and low smoke emission. This is shown graphically in Figure III-1 and the compounds' compositions are shown in Table III-11.

TABLE III-10

FULLER EVALUATION

ADL Code #	127-A	127-B	127-C	127-D	127-E	127-F
Viton A	100	100	100	100	100	100
Lead Oxide	15	15	15	15	15	15
Phoscheck P-30	100					
Cab-olite P-4		100				
Asbestos			50			
Hi Sil-233				50		
Iron Oxide					100	
Hydrated Alumina						100
Maleimide [HVA-2]	4.0	4.0	4.0	4.0	4.0	4.0
Benzoyl Peroxide	4.5	4.5	4.5	4.5	4.5	4.5
Curing Temp. (°F)	270	270	270	270	270	270
Curing Time (minutes)	45	45	45	45	45	45
Burning Characteristics in 40% O ₂ and 60% N ₂						
Ignition source and time (seconds)	H ₂ -15	H ₂ -15	H ₂ -15	H ₂ -15	H ₂ -15	H ₂ -15
Total Flame Time after ignition source is removed (seconds)	B.C. 30	B.C. 40	B.C. 50	B.C. 30	B.C. 35	B.C.* 30
Total burning time including after glow (seconds)	30					More than 180
Burned length (inches)	1/2	4	4	4	4	2 1/8
NASA Test Results in 10 PSIA, 31% O ₂ & 69% N ₂						
Burning time (seconds)	75					
Burned length (inches)	1					
Smoke	Dense white smoke					

B.C. = Burned Completely

FIGURE III-1
EFFECT OF AMMONIUM POLYPHOSPHATE
AND HYDRATED ALUMINA

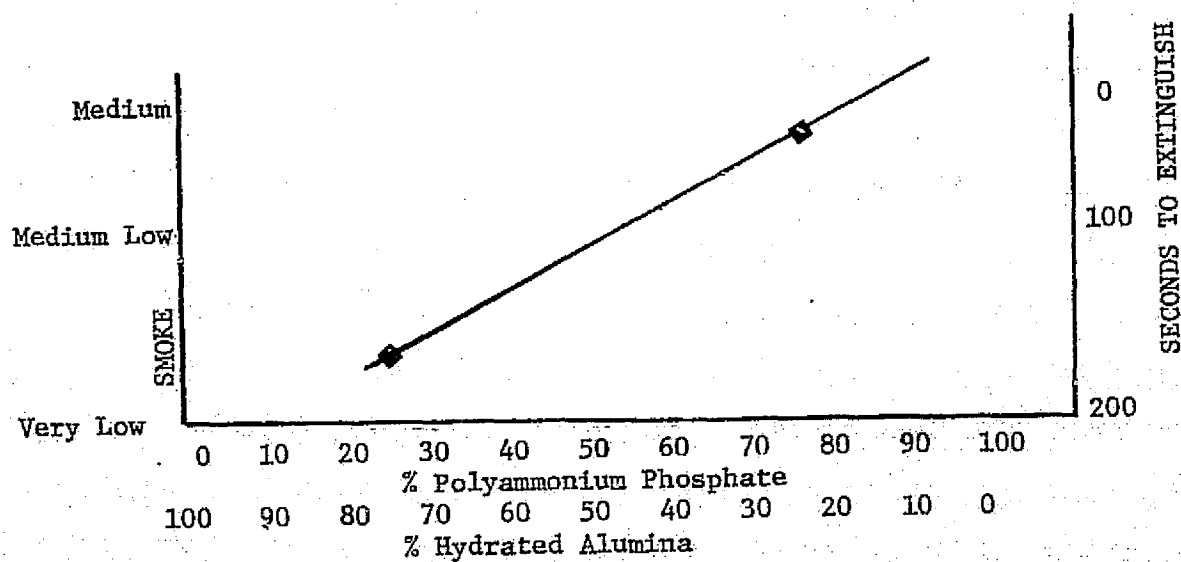


TABLE III-11
SMOKE COMMISSION EVALUATION

<u>ADL Code #</u>	<u>128B</u>	<u>128C</u>	<u>128D</u>
<u>Ingredients</u>	<u>Parts By Weight</u>		
Viton B	100	100	100
Lead Oxide	15	15	15
Polyammonium Phosphate (P-30)	25	50	50
Hydrated Alumina	75	50	50
Maleimide	4.0	4.0	--
Benzoyl Peroxide	6.0	6.0	--
Diak #3	--	--	5
Curing Temperature (°F)	270	270	300
Curing Time (Mins.)	60	60	60
NASA Test Results in 10 psia 31% O ₂ and 69% N ₂			
Burning time (Seconds)	160	110	90
Burned length (Inches)	1 1/4	3/4	1
Smoke	Medium white	Medium white	Very little

IV. PRODUCTS

During the project, we produced a variety of products to demonstrate the feasibility of manufacturing them using the new compounding technology and to show that acceptable levels of flame retardancy could be obtained. The task assigned was to produce samples of foam, coated fabrics, paints, adhesives, and hose. The first two, foams and coated fabrics, were to receive first priority, since they are required for the Space Shuttle program. Consequently we devoted the major portion of our effort to these products.

A. FOAMS

In the development of flame retardant foams, we worked with Viton and urethane systems. The Viton foams are inherently more flame resistant and require less modification to provide the flame resistance required by NASA.

1. Fluoroelastomer Foam

The starting point in developing this foam was to use a formulation recommended in the Du Pont literature, evaluate it, and modify it as necessary. This fluoroelastomer foam had a density of 25 pounds/cubic foot. It was a closed cell structure and had a limiting oxygen index of 32/34. The formulation we used in making this foam is given in Table IV-1.

The procedure used in preparing Viton (fluoroelastomer) foam formulations discussed in this section was to compound on a cool mill and prepare a preform--a little less than half of the desired finished foam thickness--which was placed in a mold. The compound was sheeted out with just enough material so that excess stock was forced out of the mold when it was closed. The exact cure time depended on the particular formulation that was used. In the above example, the material cured in 30 minutes at 350°F. When the mold pressure was released, the final expansion took place to give a compound about twice the thickness of the mold.

In the basic formulation (see Table IV-1, No. 118A), which used magnesium oxide as a curing agent, the limiting oxygen index without other ingredients was between 32 and 34. To improve the flame resistance we took several steps, which included using lead oxide curing agent (this gives inherently better flame resistance), hydrated alumina, and decabromobiphenyl oxide.

Our first step was to develop a foam formulation that would cure with lead oxide. In our preliminary work, formulations using lead oxide did not cure and we found it was necessary to use a larger quantity of Diak #1 to provide a satisfactory cure with a lead oxide system. Table IV-1 lists the formulations and the L.O.I. of various samples that were

TABLE IV-1

VITON FOAM

<u>ADL CODE #</u>	<u>118-A</u>	<u>122-A</u>	<u>122-B</u>	<u>123-A</u>	<u>123-B</u>
Viton A	100.0	100.0	100.0	100.0	100.0
Magnesium Oxide	15.0	-	-	-	-
Lead Oxide	-	15.0	15.0	15.0	15.0
Petroleum Jelly	3.0	3.0	3.0	3.0	3.0
Diak #1	1.25	1.25	2.0	-	-
Maleimide (HVA-2)	-	-	-	6.0	4.0
Benzoyl Peroxide	-	-	-	6.5	-
t-Butyl Perbenzoate	-	-	-	-	4.5
Diethylene Glycol	2.0	2.0	2.0	3.0	-
Celogen AZ	5.0	5.0	5.0	-	-
Unicel ND	-	-	-	9.0	9.0
Nitrosan	-	-	-	-	3.0
Curing Temperature (°F)	350	350	350	300	300
Cure Time (minutes)	30	30	30	30	30
Post Cure Temperature (°F)	-	-	400	-	-
Post Cure Time (hours)	-	-	4	-	-
L.O.I.	32	did not cure	40-45	did not cure	did not cure

evaluated. Formulations 123A and 123B were attempts to make a foam using a maleimide (HVA-2) curing system, but apparently the blowing agent interfered with the curing.

Du Pont produces a flame resistant Viton compound, VS-2001, which has been used in a number of applications for NASA. This compound had never been used to make a foam, but we believed it was worth evaluating to determine whether it would do so. This formulation uses lead oxide as the curing agent and is very similar to our standard Viton A formulations. When used for making a foam formulation, VS-2001 would not cure properly (see Table IV-2) and it was necessary to add more curing agent than recommended by Du Pont. Since elastomer slabs prepared from VS-2001 were not more flame resistant than lead oxide/Diak #1 cured Viton A (Table IV-3), we did not evaluate this material further.

We tried several experiments in which the cure temperature and the levels of curing agents were varied (Table IV-4). Samples of the formulation that proved satisfactory, 22-1, were submitted to NASA and are currently used in the visor for the light flashing experiment (Figure IV-1).

2. Polyurethane Foam

Unmodified polyurethane foams are quite flammable and will burn in air. The L.O.I. is normally between 19 and 22. Samples of a variety of commercially available materials marketed as flame resistant flexible foams were obtained but none had L.O.I. values above 26. One sample, made from polyols and an isocyanate and sold by Grace Chemical, was the most promising and we incorporated additives into the polyol component to improve retardancy. The same procedure was tried with a series of polyols and isocyanates from the Interpolymer Corporation and these produced good flame resistant foams. However, these foams were too dense (6-10 pounds/cubic foot) for many applications.

Another approach evaluated was the treatment of commercially available foams with flame retardants. The basic problem with the available flexible urethane foams is that they have inherently poor flame resistance and must be heavily treated or modified in order to meet NASA's requirements. However, they do have the advantage that they can be made in very low densities.

The Grace Chemical flame resistant foam is a hydrophilic polyurethane that is prepared by mixing the polyol FHP 3000 (which contains reactive polymeric isocyanates) and water in a ratio of 1:1. The mixture can then be poured into a mold to obtain any desired shape. It has a limiting oxygen index of 25-26 and thus will not burn in air. By the use of additives it was possible to raise the L.O.I. up to 40. However, we found that it was extremely difficult to simultaneously get a low density and uniform cell structure. (See Table IV-5, Sample 1210.)

TABLE IV-2

FOAM FROM DUPONT FORMULATED COMPOUND

<u>ADL Notebook #134-</u>	<u>Parts by Weight</u>		
	<u>-A</u>	<u>-B</u>	<u>-C</u>
<u>Materials:</u>			
DuPont VS-2001	100	100	100
Curing Agent (F.056)	2.0	1.5	2.5
Celogen AZ	5.0	5.0	5.0
Diethylene glycol	2.0	2.0	2.0
Mold Dimension (inches)	4 x 4 x 1/4	4 x 4 x 1/4	4 x 4 x 1/4
Molding Temp. (°F)	340	340	340
Molding Time (Min.)	30	30	30
Foam Thickness (inches)	did not cure and foam	did not cure and foam	1/2

TABLE IV-3

COMPARISON OF VITON A AND DUPONT VS 2001

<u>ADL Code No.</u>	<u>136A*</u>	<u>136B**</u>
<u>Materials</u>	<u>Parts by Weight</u>	
Dupont VS 2001	100	100
Viton A	---	100
Lead Oxide	---	15
Curing Agent (F.056)	1.1	---
Diak #1		1.25
Molding Temp. (°F)	310	310
Molding Time (Mins.)	30	30
Posture Temp (°F)	400	400
Posture Time (hours)	16	16
L.O.I.	50	50

*DuPont flame retardant compound

**ADL flame retardant compound

TABLE IV-4FACE MASK FOAM: MODIFIED FORMULATIONS

Code 18540	21-1	21-2	21-3	22-1
<u>MATERIALS</u>	<u>PARTS BY WEIGHT</u>			
Viton A	100	100	100	100
Lead Oxide	15	15	15	15
Carbon Black	2	2	2	3
Diak #1	2	2	2.25	1.75
Celogen AZ	7	7	7	5
Diethylene Glycol	2	3	2.5	2
Molding Temperature (°F)	327	320	320	340-345
Time Cure (min.)	15	15	15	35
<hr/>				
Too Hard	✓		✓	
Satisfactory				✓
Undercured		✓		

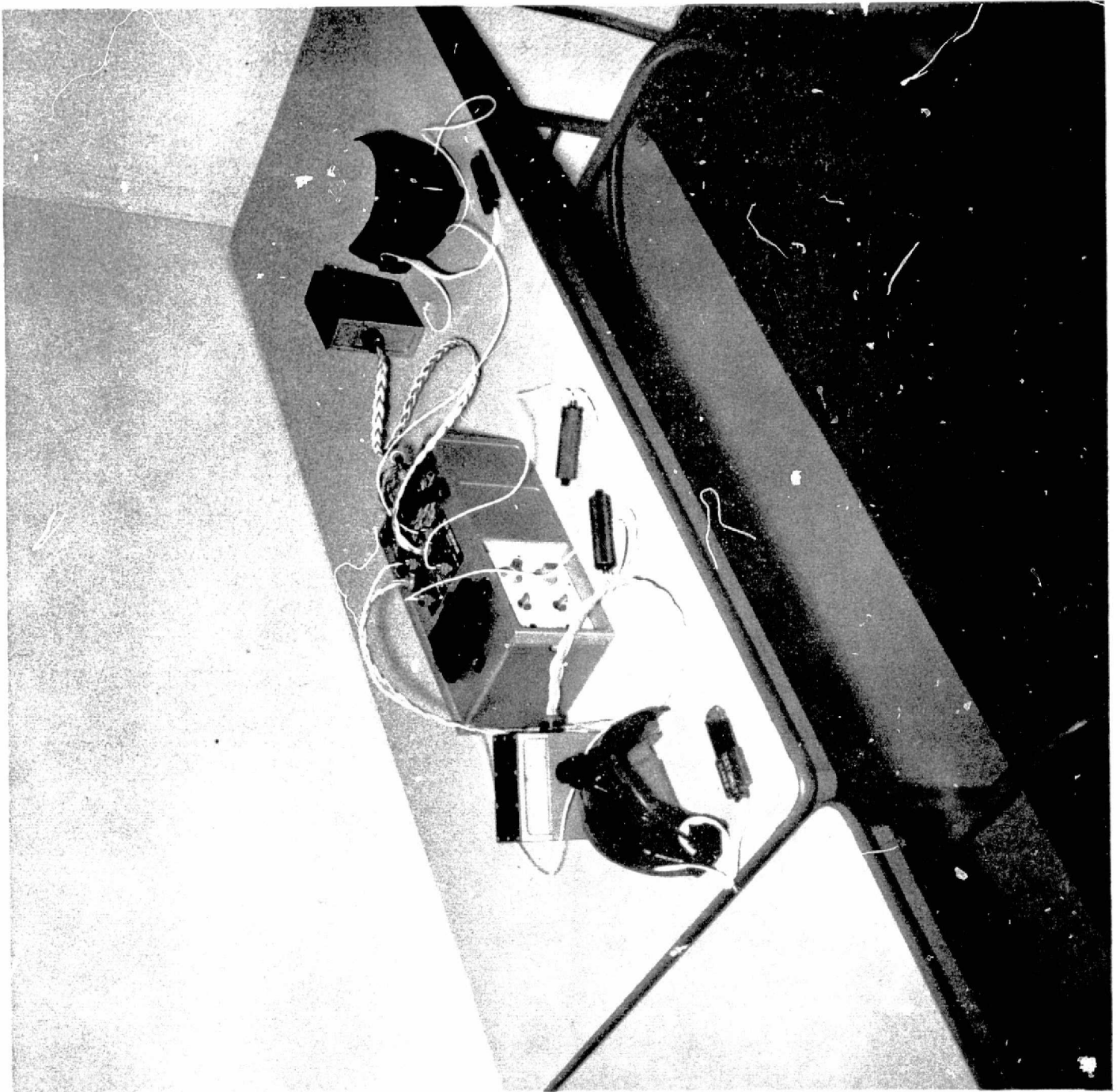


FIGURE IV-1
VITON FOAM USED ON VISOR OF
LIGHT FLASHING EXPERIMENT

TABLE IV-5

GRACE POLYURETHANE FOAM

<u>ADL CODE No.</u>	<u>121-A *</u>	<u>121-B</u>	<u>121-C</u>	<u>121-D</u>	<u>121-E</u>	<u>121-F</u>	<u>121-G</u>	<u>121-H</u>
Hypol FHP 3000	100	100	100	100	100	100	100	100
Ammonium Bromide	-	100	-	-	66	-	-	-
2,3-Dibromopropylphosphate	-	-	100	-	-	-	-	-
Hydrated Alumina	-	-	-	100	-	-	-	-
Ammonium Phosphate	-	-	-	-	33	-	-	-
Urea Formaldehyde Resin	-	-	-	-	-	100	200	300
Water	100	100	100	100	100	-	-	-
29 Comments on Physical Properties	Good Foam	Turned Yellow after 1 Wk	Next Best to Control	Semi-Rigid	Turned Yellow	Semi-Rigid	Rigid	Did Not Foam
L.O.I.	25-26	35-36	35-40	26-27	30-35	24-25	-	-

* Control-unmodified formulation

Because of the limitations of this foam, we evaluated several methods of treating urethane foam to improve its flame retardancy. Our first step was to treat polyurethane foam with a fluoropolymer solution containing flame retardants. MEK was used as the solvent but the MEK rapidly softens and causes the urethane foam to swell and therefore cannot be applied in this manner.

Our first attempt to overcome this difficulty was to laminate a Viton formulation with suitable flame retardants to a urethane foam. We spread-coated a sheet of the fluorocarbon formulation onto a release paper from a 40% solution in MEK, cured the coating in an oven at 300°F for 1 hour and post cured it at 400°F for 24 hours. A very thin coating of the same formulation in MEK was then applied to the back of the sheet and the sheet was then laminated to a Scott foam.

This lamination provided very excellent protection against flame, when the flame was applied to the coated surface. However, if the flame was applied to the edge of the sample which was not covered, the inner portion of the urethane burned rapidly. Thus, it was an impractical method of flame proofing, and saturation with a solution appeared to be the only acceptable technique.

Since MEK tended to swell the foam, we switched to acetone as the solvent.

We selected a Scott Pyrell foam which has a density of about 1.8 pounds/cubic foot and an L.O.I. of 22, and saturated samples of it with solutions containing different solids. This approach yielded foams that varied in density from 12 pounds/cubic foot to 24 pounds/cubic foot. These were sent to NASA.

A request was made for a foam with a density of 10 pounds/cubic foot in a sample 100 feet long, 18 inches wide, and 1/8 inch thick. We prepared this sample on our pilot coater. Table IV-6 gives the coating formulation and the exact conditions that were used. This foam was used as padding in and around electronic equipment (Figures IV-2 and IV-3).

The Space Shuttle program requires a method of packaging instruments, so we prepared a number of samples of various types of flexible urethane foam which could be fabricated into slabs in which holes could be made to hold the articles. As a starting point, we obtained a commercial urethane foam composition consisting of a polyol and an isocyanate and modified it with flame retardant at ADL. The retardants evaluated were decabromobiphenyl oxide, ammonium polyphosphate, and antimony oxide. The density of the modified foam was 8 pounds/cubic foot.

A lower-density sample that was somewhat more rigid was requested. Therefore, a series of new foams was prepared, based on three different compositions from Interpolymer Research Corporation. (See Table IV-7.) Each had a density of 4 pounds/cubic foot before modification.

TABLE IV-6
FOAM SATURATING SOLUTION

ADL Code No. 152

<u>MATERIALS</u>	<u>PARTS BY WEIGHT</u>
Viton B	100
Lead Oxide	15
Molybdate Orange	10
Decabromobiphenyl Oxide	100
Ammonium Polyphosphate	50
Diak #1	2.5

Coated as 30% solids in acetone.

SATURATING CONDITIONS OF PILOT COATER

Squeeze roll opening	35 mils
Coater speed	2 ft/min

<u>DRYING OVEN</u>	<u>Temperature</u> (°F)	<u>Dwell Time</u> (Minutes)
1st Zone	140	4
2nd Zone	225	4
<u>CURING OVEN</u>		
1st Zone	275	16
2nd Zone	275	16

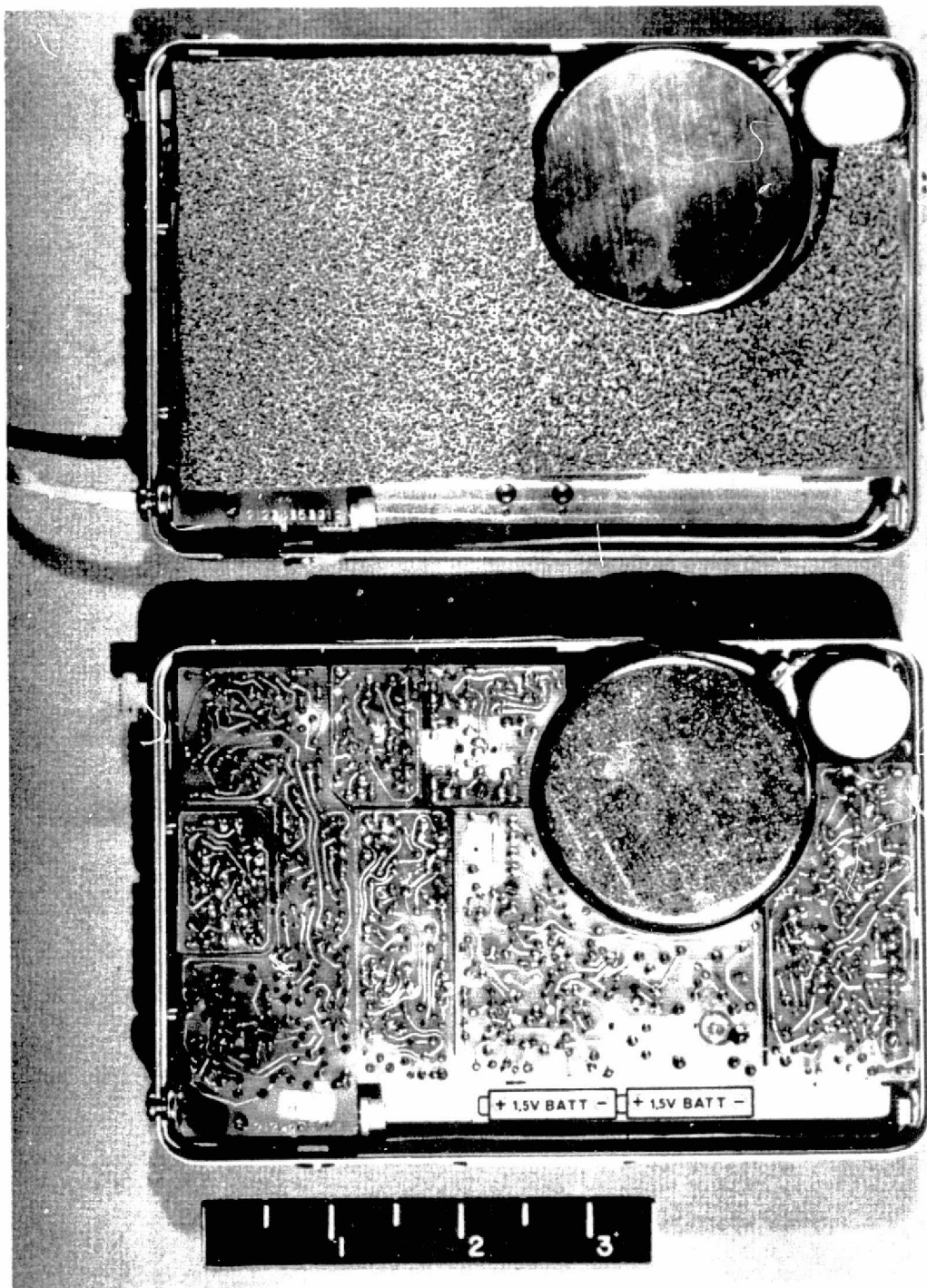


FIGURE IV-2 FLAME RETARDANT FOAM PADDING
(VITON-IMPREGNATED PYRELL FOAM)
USED AS PROTECTION FOR ELECTRONIC
COMPONENTS

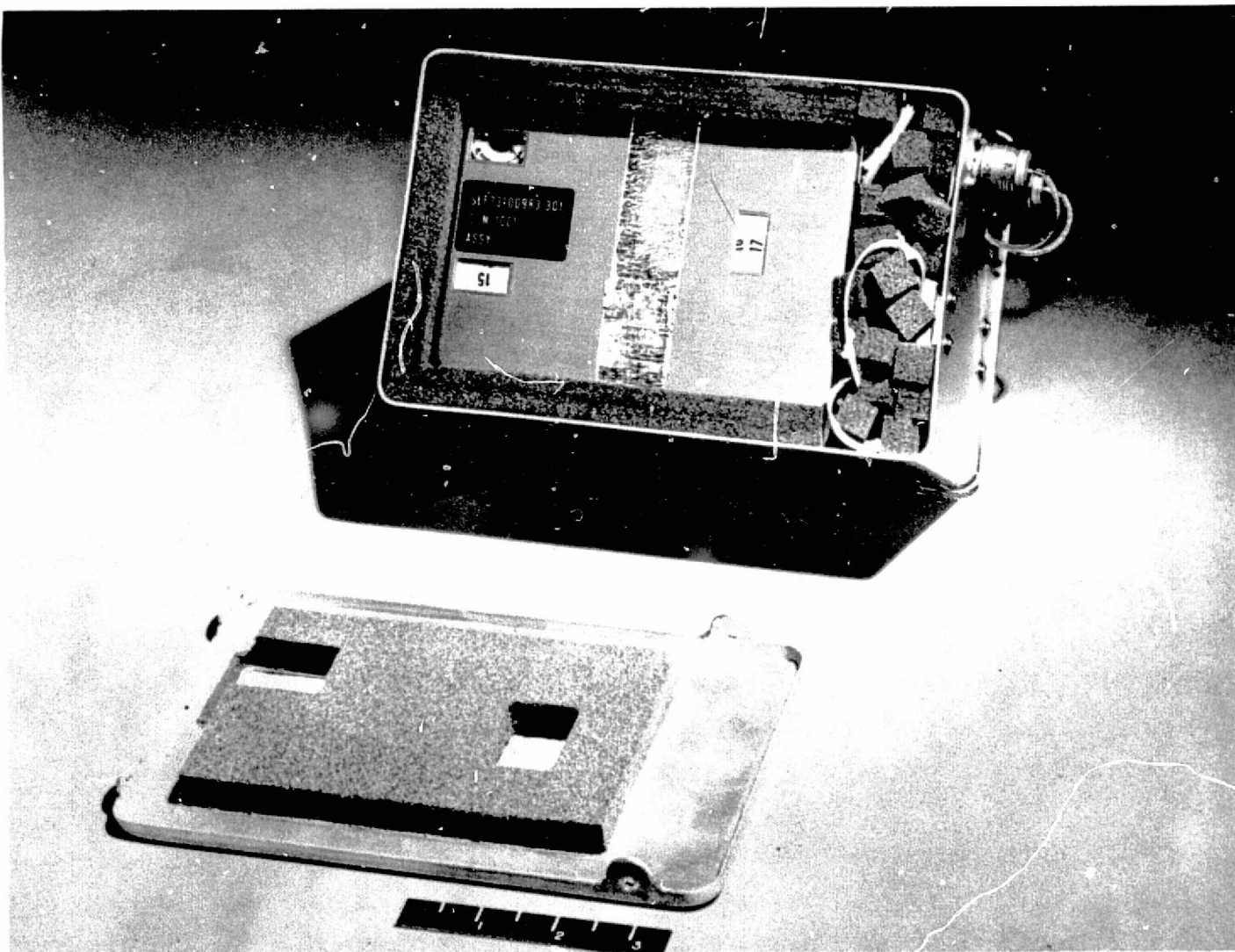


FIGURE IV-3 FLAME RETARDANT FOAM PADDING
(VITON-IMPREGNATED PYRELL FOAM)
USED AS PACKAGING FOR ELECTRONIC
EQUIPMENT

TABLE IV-7

INTERPOLYMER FOAM INGREDIENTS

Code 18540	20-1	20-2	20-3
Color	Green	Pink	Blue

MATERIALS

PARTS BY WEIGHT

Polyol FM 3603	100		
Polyol FM 3601		100	
Polyol Part A			100
Isocyanate 1112(09-73-102)	88		
Isocyanate 1612(01-74-102)		81	
Isocyanate Part B			75
Hardness (1)	3	2	1
Density (lbs/cu ft)	4	4	4

(1) Hardness rating - softest #3, hardest #1

Formulation 20-1 was selected for further modification with different levels of flame retardant. (See Table IV-8.) However, the densities of these modified foams ranged from about 5 pounds/cubic foot to 9 pounds/cubic foot, which NASA considered too dense for this application. Therefore, we went back to saturation techniques with Scott's Pyrell foam, which has a density of about 2 pounds/cubic foot.

We saturated the Scott's Pyrell foam with the composition shown in Table IV-6 which was dissolved to make a 30% solution in acetone. This yielded a foam with a density of about 3 pounds/cubic foot. We did not consider coverage satisfactory but this low level of pick-up had to be used to keep the density at the low level required by NASA.

It appears impractical, for NASA's instrument packaging, to flame-proof urethane foam by this saturation technique if the density must be less than 4 pounds/cubic foot. On this basis we concluded that the material was unsuitable for this application.

3. Ethylene Propylene Foams

A series of ethylene propylene rubber foam compounds was prepared and sent to NASA for evaluation. They all burned readily in a 31% oxygen, 69% nitrogen atmosphere. Since they showed no promise, work on them was halted. (See Table IV-9.)

B. COATINGS

Of special interest to NASA was the development of an elastomer coated fabric for use in astronauts' and firefighters' suits. The ultimate result of this program has been the coating of 50 yards of 36-inch-wide nylon rip stock with a neoprene formulation. This coated nylon is being made into suits.

In preliminary work, we used a lamination approach to prepare coated fabric samples because this method minimizes penetration of the elastomer into the fabric and prevents stiffening of the fabrics. In the preparation of the coated fabric the elastomer was first compounded on a two-roll mill and sheeted off at a thickness of about 1/16". This sheet was then placed over the fabric, overlaid with a sheet of embossed release paper, and the combination cured in a press. Normally, a temperature of 275°F and a pressure of 500-750 psi were used. In the first experiments two elastomers (Viton and silicone rubber) and two lightweight fabrics (Fire-Stop cotton by Cotton Incorporated, and Nomex) were used. The formulations that were used are shown in Table IV-10.

Both the fabric coated with Viton and that coated with silicone were self-extinguishing in a 10-psi atmosphere at 31% oxygen, 69% nitrogen. The urethane formulation burned totally. The silicone, urethane and Viton (in this formulation) gave off a dense smoke whereas the Viton formulation was modified with hydrated alumina and ammonium polyphosphate and gave off only a small amount of white smoke.

TABLE IV-8

INSTRUMENT PACKAGING FOAMS

Code 18540	23-1	23-2	23-3	23-4	23-5
<u>MATERIALS</u>	<u>PARTS BY WEIGHT</u>				
Polyol CTF	150	150	150	150	150
Isocyanate	126	126	126	126	126
Decabromobiphenyl Oxide	112.5	84.4	56.3	28.1	-
Phoscheck P-30	112.5	84.4	56.3	28.1	-
Antimony Oxide	50	25	12.5	7.5	-
2,3-Dibromopropylphosphate	30	22.5	15	7.5	-
Percent Flame Retardant	110	83	55	28	0
Density (lb/cu ft)	8.86	7.74	6.58	5.03	4.16

TABLE IV-9

FOAMETHYLENE PROPYLENE TERPOLYMER (NORDEL)

<u>Material</u>		<u>Parts By Weight</u>		
<u>Code</u>	<u>107</u>	<u>A</u>	<u>B</u>	<u>C</u>
Nordel 1040		100	100	100
Zinc Oxide		5	5	5
Stearic Acid		10	10	10
Whiting		60	60	60
Clay		50	50	50
Hydrated Alumina (Alcoa C-333)		110	110	110
Light Process Oil		100	50	50
Ammonium Bromide		-	80	80
Sodium Bicarbonate		20	20	20
Celogen AZ		0.5	0.5	0.5
MBT		0.5	0.5	0.5
Thiuram M		1.0	1.0	1.0
EPTAC 1		1.0	1.0	1.0
Tellurium Diethyl Dithiocarbamate		1.0	1.0	1.0
Ferrocene FE 55		-	-	4.0
Sulfur		4.0	4.0	4.0
Cure Condition minutes @ 350°F		30	30	30
L.O.I.		20-21	28	28
Smoke		Very low	Low	Low
<u>NASA Tests</u> *				
Burning Time (seconds)		Total burn	Total burn	Total burn
Burned Length (inches/second)		.38	.1	.166
Smoke ¹				
Density		Heavy	Dense	Dense
Color		White	Black	Black
Odor		odor	-	

* Test atmosphere 10 psia, 31% O₂, 69% N₂
Silicone ignitor

¹ Arthur D. Little, Inc., comment. The flame and smoke on these samples are much like those of a candle. Light color and substantially less smoke than most polymers give off.

TABLE IV-10

COMPOUNDS USED ON COATED FABRICS

ADL Code No. Coated Fabric	<u>Viton</u>		<u>Silicone</u>		<u>Urethane</u>	
	116A (Cotton)	116B (Nomex)	116C (Cotton)	116D (Nomex)	117A (Cotton)	117B (Nomex)
Compound Code No.	118-A		118-C		119-A	
Viton B	100.0		-		-	
G.E. SE 517	-		100.0		-	
Estane 58370	-		-		100.0	
Hydrated Alumina	100.0		-		100.0	
Decabromobiphenyl Oxide	-		75.0		-	
Lead Oxide	15.0		-		-	
Ferric Oxide	5.0		2.0		-	
HVA-2	4.0		-		-	
Benzoyl Peroxide	4.5		-		-	
2,4-Dichlorobenzoyl Peroxide (50% active in Silicone Oil)	-		1.2		-	
Curing Temperature (°F)	270		270		350	
Cure Time (minutes)	45		15		5	
Post-Cure Temperature (°F)	-		300		-	
Cure Time (minutes)	-		90		-	

Samples of this formulation, colored and coated on fabrics, were submitted to NASA for preliminary testing and examination. These were prepared as described previously, by spraying the coating onto embossed paper, applying a bond coat to the fabric, and laminating the two together and curing. The modified Viton formulation is shown in Table IV-11.

We also evaluated Kevlar, a new modified polyamide fabric, for use as a carrier for the flame resistant coatings. Kevlar is particularly well adapted for this use since it has four times the strength of nylon and has an L.O.I. of 30. We coated a 7-oz Kevlar felt, and 5- and 2.2-oz fabric.

In this set of experiments, two methods of curing were used. For heavier coatings, a calendered sheet was press-cured directly onto the Kevlar fabric and for lighter coatings a 40% solution in MEK was spread-coated onto embossed release paper. Several layers of coatings were applied until a suitable thickness was achieved. The coating was then dried, a coat of Viton solution applied to the Kevlar, and the two surfaces laminated together in a press and cured in a 310°F oven for 2 hours. The formulations and processing methods are shown in Table IV-12. These samples did not burn in a 31% oxygen, 69% nitrogen atmosphere.

Since one of the major use potentials was in firefighters' suits, samples were prepared using a chrome yellow or molybdate orange to give the desired appearance. The formulations that were used are shown in Table IV-13.

C. COATED FABRICS

A series of coated fabrics was prepared using different coating formulations on different types of fabrics to achieve the desired properties. Table IV-14 lists the formulations that were evaluated, the cost and the oxygen atmosphere in which they will not burn. Formulations 1 and 2 are based on Viton and decabromobiphenyl oxide and are suitable for a 70% oxygen atmosphere. Since decabromobiphenyl oxide is used, the material does produce some smoke when ignited. Formulation 3, which is based on hydrated alumina and ammonium polyphosphate, is satisfactory for the 30% oxygen atmosphere and creates no smoke. Formulations 4 and 5 are urethane formulations with different levels of flame retardancy to meet the requirements of non-flammability in lower oxygen atmospheres.

Firefighters' suits would probably best be made using Kevlar or Nomex fabrics coated with either Viton, urethane or neoprene compositions. Fabric weighing about 2 1/2 ounces appears to be the most suitable for firefighters' suits. The most suitable formulations for this purpose are the Viton formulations (Table IV-14) or the urethane formulation (Formulation 4, Table IV-14).

TABLE IV- 11
LOW SMOKE COMPOUND

<u>Material</u>	<u>Parts By Weight</u>
Viton B	100
Lead Oxide	15
Polyammonium Phosphate [Phoscheck P-30]	50
Hydrated Alumina	50
Pigments Either blue (Monastral Fast Blue BC DuPont) or red (Iron Oxide)	5
Diak #3	4

TABLE IV-12

COATED KEVLAR FORMULATIONS

<u>ADL Notebook No.</u>	<u>137A</u>	<u>137B-1</u>	<u>137B-2</u>	<u>137B-3</u>	<u>137B-4</u>	<u>137B-5</u>	<u>137B-6</u>
<u>Materials</u>	<u>Parts by Weight</u>						
Viton B	100	100	100	100	100	100	100
Lead oxide	15	15	15	15	15	15	15
Hydrated alumina	50	100	100	100	100	100	100
Phoschek P-30	50	--	--	--	--	--	--
Iron oxide	5	5	5	5	5	5	5
Diak #3	4	4	4	4	4	4	4
Type of Fabric							
Kevlar Felt, 7 oz						x	x
Kevlar, 5 oz	x	x	x	x			
Kevlar, 2.2 oz					x		
Technique used in applying coating	press	press	SC*	SC*	SC*	SC*	SC*
Thickness of coating (mils)	10	10	5	3	3	5	3
Curing Conditions							
Temperature (°F)	310	310	310	310	310	310	310
Time (Hours)	1/2	1/2	2	2	2	2	2

* Spread coating

TABLE IV-13
COATED FABRICS

<u>ADL Notebook No.</u>	<u>142A</u>	<u>142B</u>
<u>MATERIALS</u>	<u>PARTS BY WEIGHT</u>	
Viton B	100	100
Lead oxide	15	15
Hydrated Alumina	50	50
Ammonium Polyphosphate	50	50
Chrome Yellow	15	--
Molybdate orange	--	15
Diak #1	2.0	2.0

Note: Six samples, three of each formulation, 3-mil thickness on 2-oz Kevlar fabric and 7-oz Kevlar felt, and 5-mil thickness on 5-oz Kevlar were prepared and sent to NASA.

TABLE IV-14

COATED FABRIC FORMULATIONS

Material	Parts by Weight				
	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>
Viton	100	100	100	---	---
Lead Oxide	15	15	15	---	---
Diak #1	2	2	2	---	---
Decabromobiphenyl oxide	100	50	---	---	100
Hydrated Alumina	---	---	50	50	---
Phoschek P-30	---	---	50	50	---
Color	5	5	3	3	3
Urethane	---	---	---	100	100
Methyl Ethyl Ketone	---	---	---	---	---
Cost/pound (\$)*	7.2	8.08	6.02	1.07	1.55
Weight/Mil/yd (lb)	.1104	.1082	.0954	.0687	.0776
Cost/Mil/sq yard coating	.794	.874	.574	.075	.121
Cost 6-mil coating 2/sides (\$)	4.76	5.25	3.45	0.45	0.72
For oxygen atmosphere					
20 - 30				x	
Over 30			x		x
Over 70	x	x			
Fabric Cost with 15% loss (2 1/2 oz fabric)					
Kevlar	6.75	6.75	6.75	6.75	6.75
Nomex	5.00	5.00	5.00	5.00	5.00
Glass	0.63	0.63	0.63	0.63	0.63
Dacron	1.50	1.50	1.50	1.50	1.50
Estimated coating cost/yd for 25 yds	13.00	13.00	13.00	13.00	13.00

* Includes solvent cost

Purge ducts are best made using a Kevlar coated with formulation. Table IV-15 lists coating formulations that might be used, and the cost per mil per square yard for Viton and urethane compositions. Fabric costs for Kevlar, Nomex and Dacron 2 1/2-oz fabric are also shown. Quotations for coating 25 yards of material averaged about \$13.00 per square yard. Reeves Brothers quotes \$325 for setup and coating charges for 25 yards or less, and Du Pont quotes prices of \$1,000 to \$5,000 for Viton formulations. Du Pont supplies the materials. Table IV-15 shows the costs of the coating materials that have been used in the estimates and the per yard cost for 38" and 50" wide fabric.

Table IV-16 provides an estimate of the costs of coating the formulations shown in Table IV-14 if the coating is 3 mils thick on each side. The coating charges have been estimated at \$15.00 per square yard for a small quantity. This would automatically drop under production conditions. As shown in Table IV-16, the major item of cost with these small quantities, is the coating charge.

As part of this program to improve coated fabrics for use in the Space Shuttle program and for firefighters' suits, we also prepared fabrics with a flame resistant neoprene coating. (See Table IV-17.)

Two nylon fabrics, one blue and one white, sent to us by NASA were used. The sample code was 18540-3A for the blue nylon and 18540-3B for the white nylon. In each case the coating formulation was the same and the coating thickness was between 4 and 5 mils.

In addition, a sample of a flame resistant Viton formulation was coated on 2.2-oz Kevlar fabric from a 40% solution in MEK. Four coatings were applied to each side with a No. 40 Myer rod and this resulted in a 3- to 4-mil coating on each side. The samples were cured in an oven at 200°F for 2 hours, and then post-cured at 325°F for 3 hours. These samples were approved by NASA for further trials and the production of quantities of material if required. The formulation is shown in Table IV-18.

Because of cost, ease of application, and high level of flame retardancy, the neoprene formulation on nylon rip stop was the most interesting of all the coated fabrics that were prepared. Since samples were too large to be easily prepared on our equipment, we arranged to do the actual coating at a plant in Connecticut operated by Du Pont's Fabrics & Finishes Division. They first prepared panels of the material about 2 yards long. These were sent to NASA and approved. Later a coating run was made and 50 yards of coated fabric was prepared. This sample is being constructed into prototype space suits by the ILC Corporation in Dover, Delaware, and thermal gloves by the Acurex Corporation in California. The final product weighed 6 ounces/square yard and used a 4- to 5-mil coating on the nylon. The coating was applied at a weight of 5 inches/square yard.

TABLE IV-15
COST OF MATERIALS

<u>Material</u>	<u>\$/lb</u>
<u>Coating</u>	
Viton A, B, and A-35	10.00
Viton G-72	10.50
Diak #1	13.75
Diak #3	5.25
Decabromobiphenyl oxide	2.27
Hydrated Alumina	.07
Phoschek P-30*	.67
Lead Oxide	.20
Color	.67
Urethane	1.00
<u>Fabrics</u>	<u>Dollars/Running Yard</u>
1 - Fiber Glass, beta type	<u>38-inch width</u> <u>50-inch width</u>
2.6 oz	0.65 0.86
5 oz	
1 - Kevlar	
2 1/2 oz (Style 220)	6.75 7.70
5 oz (Style 281)	6.95 7.90
2 - Nomex	
2 1/2 oz	5.00 6.50
3 - Dacron	
3 oz	1.50

*Ammonium Polyphosphate

TABLE IV-16

PRODUCT COSTS

(\$/square yard based on coating 25 square yards)

<u>Composition</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>
1. Coating Material 2-side 3 mil coating	4.76	5.25	3.45	0.45	0.72
2. Coating Charge	13.00	13.00	13.00	13.00	13.00
3. Fabric Cost 2 1/2 oz., 40" wide Kevlar	6.75	6.75	6.75	6.75	6.75
Grand Total Kelvar (1 + 2 + 3)	24.51	25.00	23.20	20.20	20.47
4. Nomex	5.00	5.00	5.00	5.00	5.00
Grand Total Nomex (1 + 2 + 4)	22.76	23.25	21.45	18.45	18.72
5. Glass	0.63	0.63	0.63	0.63	0.63
Grand Total Glass (1 + 2 + 5)	18.39	18.88	17.08	14.08	14.35
6. Dacron	1.50	1.50	1.50	1.50	1.50
Grand Total Dacron (1 + 2 + 6)	19.26	19.75	17.95	14.95	15.22

ALL MATERIAL COSTS ARE BASED ON A 15% WASTE ALLOWANCE.

TABLE IV-17

COATED FABRICS

<u>Materials</u>	<u>Parts by Weight</u>
Neoprene W	100
Magnesium Oxide	4
Akroflex CD	2
Hydrated Alumina	33-1/3
Ammonium Polyphosphate	33-1/3
Decabromobiphenyl Oxide	33-1/3
Antimony Oxide	15
Zinc Oxide	5
Carbon Black	2
NA-22	1

Solvent M.E.K., 4 parts to 1 part toluene—as a 40% solution.

TABLE IV-18

KEVLAR COATING FORMULATION

<u>Materials</u>	<u>Parts by Weight</u>
Viton B	100
Lead Oxide	15
Chrome Yellow	10
Hydrated Alumina	50
Ammonium Polyphosphate	50
Diak #1	2

Solvent M.E.K.--as a 40% solution.

D. FILM AND SHEETING

Film and sheeting can be prepared by casting, extrusion, and calendering. Film and sheeting may be made from the same compositions that were used for coated fabric--fluorinated elastomer (Viton), neoprene or urethane. In order to illustrate that films could be made of the material, we worked with a Viton composition that had been used for coated fabrics and spread coated or cast it onto embossed sheet, dried and cured it in an oven, then stripped it from the embossed sheet. A sheet of this material was submitted to NASA for test; it was found to have a tensile strength of about 1200 psi and an elongation of 150%. It would not burn at 10 psi in a 31% oxygen, 69% nitrogen atmosphere.

Table IV-19 shows three formulations that were evaluated. Each of them had roughly the same properties. From the standpoint of minimum smoke and optimum flame resistance, formulation 128D was the best. If color is required, pigments can be added to this formulation.

It became apparent that this same solution could also be used as a basic composition for use in paints and adhesives. Therefore, we sent samples of thin and thick films of the formulation to NASA and also a sample in which the formulation was coated onto fabric. Although these particular samples used a benzoyl peroxide cure, Diak #3 will give equivalent properties. (See Table IV-20.) Formulation 128A is the preferred formulation.

E. PAINTS

Very satisfactory paints can be prepared using the same basic formulation that was used to spread-coat the coated fabric (see Table IV-21). We prepared paints and evaluated their properties on steel panels. The steel panels were prepared by sandblasting the metal surface and then degreasing with trichloroethylene. The coatings were then applied, and put through a standard curing cycle of 3 hours at 320°F. These samples were tested at NASA for wear characteristics. In 1000 cycles, they showed a weight loss of only 0.58 gram.

A series of paints was tested on steel panels at different coating thicknesses. The abrasion resistance appears to be directly proportional to the thickness of the coating, as shown in Table IV-21. With the thin coats, the test shows the effect of substrate rigidity, which magnifies abrasion. Thus, thin coatings show poor abrasion resistant properties.

With fluoroelastomer paints, it is desirable to provide a system that will cure at room temperature. Standard formulations do not.

Fluorelastomers are vulcanized by eliminating hydrogen fluoride, which in turn provides unsaturation for cure. A standard cure is initiated by basic materials, such as Diaks, or by peroxides, such as benzoyl peroxide.

TABLE IV-19FILM AND SHEETING

ADL Notebook No.	128B	128C	128D
Viton B	1	100	100
Lead Oxide	15	15	15
Hydrated Alumina	75	50	50
Ammonium Polyphosphate [Phoscheck P-30]	25	50	50
Maleimide	4	4	---
Benzoyl Peroxide	6	6	---
Diak #3	---	---	5
Curing Time [minutes)	60	60	30
Curing Temperature [°F]	270	270	300
Post Curing Time [hours]	4	4	4
Post Curing Temperature [°F]	325	325	325
Tensile Strength [psi]	1400	1000	1200
Elongation [%]	125	150	150

TABLE IV- 20

FLAMMABILITY AND SMOKE TESTS*

<u>ADL Code</u>	<u>129B</u>	<u>129A</u>	<u>128A</u>
<u>Ingredients</u>	<u>Parts by Weight</u>		
Viton B	100	100	100
Lead Oxide	15	15	15
Polyammonium Phosphate [Phoscheck P-30]	25	25	50
Hydrated Alumina [Alcoa C-330]	75	75	50
Maleimide [HVA-2]	4.0	4.0	4.0
Benzoyl Peroxide	6.0	6.0	4.5
Thickness (inches)	0.005	0.023	0.025
NASA Test Results in 10 psia at 31% O ₂ and 69% N ₂			
Burned Length (inches)	1 1/4	Entire Sample	3/4
Burning Time (seconds)	80	50	85
Propagation Rate (cm/sec)	Self Ext.	0.1	Self Ext.
Smoke	Very light smoke	Medium Black Smoke	Light Smoke

SAMPLE DESCRIPTION:

129-B	Embossed Viton film (by solution coating)
129-A	Viton coated nylon backed fabric (by solution coating)
128-A	Viton Rubber sheet (by press molding)

* Tested at NASA

TABLE IV-21

<u>PAINTS</u>		<u>Parts by Weight</u>	
<u>Material</u>			
Viton B	100		100
Lead Oxide	15		15
Polyammonium Phosphate [Phoscheck P-30]	50		50
Hydrated Alumina C-330	50		50
Monstral Blue BC	5		--
Iron Oxide	--		5
Diak #3	4		4

Paints Abrasion Resistance Tests¹

<u>ADL Code</u>	<u>130A</u> <u>(1)</u>	<u>130A</u> <u>(2)</u>	<u>130B</u> <u>(1)</u>	<u>130B</u> <u>(2)</u>
Thickness of Coating (inches)	2.3×10^{-3}	3.6×10^{-3}	7.4×10^{-3}	5.8×10^{-3}
<u>Wear:</u>				
Weight Loss (gms/cycle)	1.1×10^{-3}	0.97×10^{-3}	0.58×10^{-3}	0.78×10^{-3}
Thickness Loss (inches/cycle)	2.2×10^{-3}	1.5×10^{-5}	0.53×10^{-5}	1.03×10^{-5}
Abrasion Resistance Cycle	58	151	750	360

Note: The above testing was done on the Taber Abraser using H-22 wheels and 500 gram weights.
The H-22 abrasive wheels simulate the worst possible wear conditions.

¹ Tested at NASA

The Diaks provide a slow cure--30 and 60 minutes at 250 and 300°F, respectively. To determine whether any cure could be obtained at room temperature, we prepared formulations containing Diak #1 and Diak #3. Both were partially cured after seven days. The formulations are shown in Table IV-22.

We also evaluated peroxides alone and a benzoyl peroxide-maleimide curing system. No cure occurred with either system in two weeks. We also examined peroxides which are active at low temperature (e.g., methyl ketone peroxide) in the presence of cobalt naphthanate. However, these also failed to cure the Viton at room temperature.

Further investigation determined that CPON H-2, a blocked amine sold by Shell Chemical, will produce a room-temperature cure. We found that the material is on strict allocation, but Shell agreed to release some directly to NASA. The room-temperature curing agent is used in place of the Diaks in a ratio of 1 part to every 100 parts of Viton.

F. ADHESIVES

For adhesive work we have used polymer systems based on the fluorinated elastomer and neoprene. The fluorinated elastomer adhesives were prepared by using a flame retardant system based on the ammonium polyphosphate and alumina, or one based on a decabromobiphenyl oxide, depending on the level of flame resistance required. The decabromobiphenyl oxide system was used in applications which required non-burning in up to a 100% oxygen atmosphere, and the hydrated alumina and ammonium polyphosphate were used in applications which required resistance to no greater than a 70% oxygen, 30% nitrogen atmosphere. Both of these systems may be cured at room temperature using the Shell H-2 blocked amine curing agent.

Before bonding the metal, we sandblasted it and cleaned it with trichloroethylene. Shear strengths on the bonded samples averaged about 250 pounds per inch. Because the bonds are flexible, they satisfactorily withstood hot and cold cycling (-40 to 400°F). Test results reported to us by NASA are shown in Table IV-23.

Adhesives based on Viton formulations have been made up at concentrations varying from about 25% solids to about 40% solids in either MEK or acetone, depending upon the application. Adhesives based on this material were evaluated at NASA for bonding metal to urethane foam and to fluoroelastomer foam, and to bond Velcro to camera lens covers.

For bonding neoprene-coated fabrics, a suitably flame-retarded neoprene adhesive was developed. The formulation used is shown in Table IV-24.

TABLE IV-22

ROOM TEMPERATURE CURE SYSTEMS

	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>
Viton B	100	100	100	100	100	100
Lead Oxide	15	15	15	15	15	15
Polyammonium Phosphate [Phoscheck P-30]	50	50	50	50	50	50
Hydrated Alumina	50	50	50	50	50	50
Pigment - Monastral Blue B.C.	--	--	--	5.0	5.0	5.0
Benzoyl Peroxide	6.0	6.0	--	--	--	--
Maleimide	4.0	4.0	--	--	--	--
Diak #1	--	--	--	2.0	--	--
Diak #3	--	--	--	--	4.0	--
MEK Peroxide	--	2.0	3.0	--	--	--
Cobalt Napthanate	--	1.0	1.5	--	--	--
Triethylene Tetramine (TETA)	--	--	--	--	--	2.0
COMMENT ON CURE						
After 1 day *	DNC	DNC	DNC	DNC	DNC	DNC
After 7 days *	DNC	DNC	DNC	PC	PC	DNC

* DNC = DID NOT CURE

* PC = PARTIALLY CURED

TABLE IV-23
METAL BOND STRENGTH*

<u>ADL Notebook No.</u>	<u>130B</u>
<u>Material</u>	<u>Parts by Weight</u>
Viton B	100
Lead Oxide	15
Polyammonium Phosphate [Phoscheck P-30]	50
Hydrated Alumina [Alcoa C-330]	50
Iron Oxide	5
Diak #3	4.0
Test Results	
Average Shear Strength (lb/in ²)	250
Range (lb/in ²)	112-390

* Tested at NASA

TABLE IV-24

NEOPRENE ADHESIVE

(40% Solids in MEK/Toluene, 4/1)

<u>Materials</u>	<u>Parts by Weight</u>
Neoprene	100
Magnetite D	4
Zinc Oxide	5
Carbon Black	4
Akroflex	2
D.B.B.P.O.	16 2/3
Hydrated Alumina	16 2/3
Phoscheck P-30	16 2/3
Antimony Oxide	8 1/3
NA-22	1

G. CAMERA LENS COVERS

In response to a request from NASA for a compound that would be flexible as well as very flame retardant for use in molding a lens cover, we prepared a number of compounds in which varying levels of flame retardant were used. While our work was in progress, the mold was being prepared at NASA and specifications were needed on shrinkage during cure so this shrinkage could be taken into account in making the mold. The compositions and hardness are shown in Table IV-25. Samples of these compounds were tested at White Sands and all were approved. Tests showed that the compound changes produced the following effects:

- Fluorinated elastomers without fillers and without plasticizer, on curing, gave a relatively hard compound of about 50 Shore A. In order to provide compounds of lower hardness, one normally adds plasticizer, but in this application it is impractical because of the high level of flame retardancy required. Plasticizers reduce flame resistance.
- As flame-retardant levels were increased, hardness increased, shrinkage during cure decreased, and levels of curing agent had to be increased to maintain adequate physical properties.
- All of the compounds extinguished in a 100% oxygen atmosphere but the time to extinguish decreased as the amount of decabromobiphenyl oxide or ammonium polyphosphate and hydrated alumina was increased.
- Less molding pressure was required and mold flow was increased as the amount of flame retardant was decreased.
- Samples that contain ammonium polyphosphate and hydrated alumina as the flame retardant must be cured and post-cured at temperatures below 340°F. Above 340°F, alumina gives off its water of hydration and the samples have a porous and brittle structure. For this type of sample we recommend that the cure and post-cure temperature be kept below 325°F. The samples selected by NASA were compounds 11C and 14B. Twenty pounds of 14B and 12 pounds of 11C were shipped to NASA. The curing agent was added to the compound at NASA to prevent precure during shipment and storage. Examples of lens caps are shown in Figure IV-4.

After a lens cover is molded, it must be attached to a piece of Velcro by means of an adhesive. An adhesive based on fluorinated elastomer was prepared and shipped. The formulation is shown in Table IV-26.

TABLE IV-25

CAMERA LENS COVER FORMULATIONS

Code 18540	13A or 11A	11C	14A	14B	13B or 14C	18B
<u>MATERIALS</u>	<u>PARTS/WEIGHT</u>					
Viton A	100	100	100	100	100	100
Lead Oxide	15	15	15	15	15	15
Carbon Black	3	3	3	3	3	3
Decabromobiphenyl Oxide			25	50	100	25
Hydrated Alumina		50				25
Ammonium Polyphosphate		50				25
Diak #3	3	6	3	3		4.5
TOTAL	121	224	146	171	222	198.5
Parts Diak #3 per 100 parts compound	2.54	2.75	2.10	1.79	1.83	1.93
Hardness Shore A	50	90-95	65	80	85-90	90

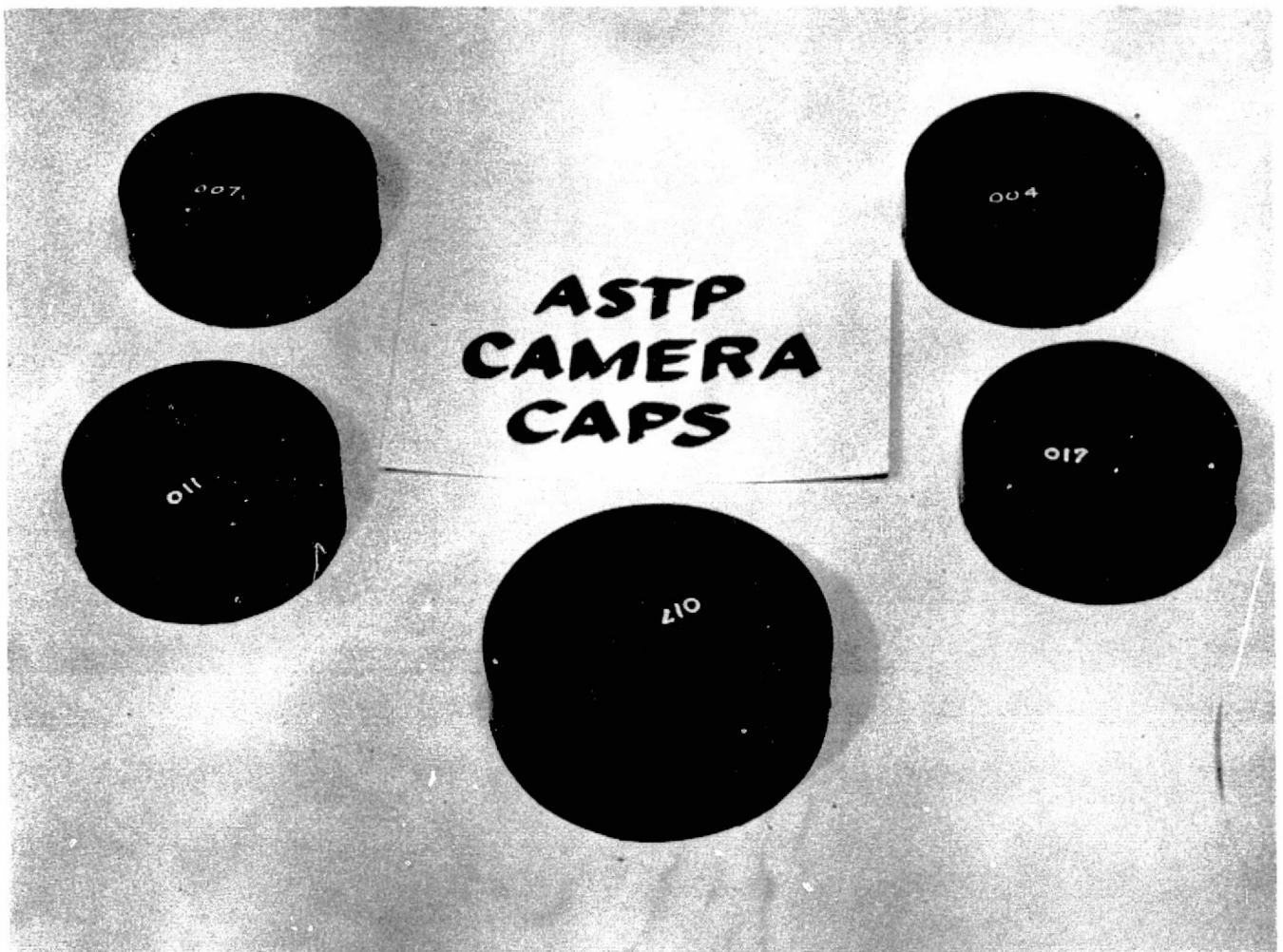


FIGURE IV-4
CAMERA LENS CAPS PREPARED
FROM VITON FORMULATION

TABLE IV-26

BONDING ADHESIVE FOR LENS COVERS

Code 18540

19A

MATERIALS

PARTS BY WEIGHT

Viton A

100

Lead Oxide

15

Carbon Black

2

Curing agent to be added Epon H-1

1

Solvent MEK - at 25% solids

H. TUBING

Twenty-five feet of 5/8" OD tubing with a 3/32" wall was extruded using a 3/4" diameter extruder with a tubing die. The objective of this work was to show that the formulation, which had been previously approved by NASA because it met the smoke and non-flammability requirements, could be extruded into tubing. The exact procedure used for making the tube was as follows:

The formulation was compounded on a 2-roll mill, ground into pellets, and charged to a 3/4" diameter extruder equipped with a tubing die. The first zone was cold; the second zone was at 150°F, and the die at 200°F. The tube was extruded at a rate of about 50 feet/hour, but faster rates are possible. The tubing was collected in coil form and then cured in an air-circulating oven as follows:

200°F for 4 hours

250°F for 12 hours

300°F for 4 hours

This formulation, which is shown in Table IV-25, has been qualified at White Sands.

I. ASBESTOS FOAM

An asbestos foam developed by the Rex Asbestwerke in West Germany is very weak and needed a spray coating on the surface to hold it together and to prevent external damage. A coating was developed that could be sprayed onto the surface and provide the desired protection. The formulation that proved satisfactory was based on the fluorocarbon elastomer given in Table IV-27.

J. TOXICITY EVALUATION

Samples of compositions for products planned for use by NASA were prepared and sent to Dr. John Antian, Director of the Materials Science Toxicology Laboratories at the University of Tennessee for toxicity evaluations. These samples (see Table IV-28) consisted of 400 grams each of slab stock based on formulations that would be used in sheeting and coating work. Included also were samples of Viton and urethane foam. All of these compositions were approved.

TABLE IV-27

ASBESTOS FOAM COATING

<u>Material</u>	<u>Parts by Weight</u>
Viton A-35	100
Lead Oxide	15
Decabromobiphenyl Oxide	100
Molybdate Orange	10

TABLE IV-28

SAMPLES FOR TOXICITY TESTS

ADL Notebook No.	150-A	150-B	151-B
<u>MATERIALS</u>	<u>PARTS BY WEIGHT</u>		
Viton A	100		
Viton B		100	100
Magnesium Oxide	15	15	15
Decabromobiphenyl Oxide	100	50	100
Diak #1	1.5	1.75	2.5
Celogen AZ		7.0	
Diethylene Glycol		2.0	
Form of Material	Flame resistant sheet	Flame resistant foam	Scott saturated foam